GAS-SELECTIVE PERMEABLE MEMBRANE AND METHOD OF MANUFACTURING THEREOF

This application claims Paris Convention priority of Italian application no. TO2003A000032 filed on January 24, 2003.

BACKGROUND FO THE INVENTION

The present invention relates to a gas-selective permeable membrane, particularly for leak detectors, and to the method for its manufacturing.

In the field of leak detection in ducts, tanks etc., the use of apparatuses known as "leak detectors" is widespread. Such apparatuses generally comprise a vacuum-tight chamber equipped with a selective membrane through which only a predetermined gas can flow into the chamber, when the pressure inside the chamber is made significantly lower than the outside pressure.

The membranes of the known leak detectors are generally made of quartz or glass with high silica content. Such membranes are permeable to helium if they are brought to a suitable temperature, typically at least 300°C. Use of such membranes has become particularly popular also because helium is a harmless, inert gas that is present in very small amounts in the atmosphere and hence is suitable for use as a test gas for leak detection.

An electrical resistor is generally used to bring the membrane to the temperature at which the membrane material becomes permeable.

The operation of the leak detectors is as follows: once a sufficient vacuum has been created in the chamber, the detector can absorb, through the selective membrane, an amount of the test gas. If the test gas is present in the surrounding environment, for instance because of a leak from a volume into which said gas has been previously introduced, the gas penetrates into the detector chamber from which it is pumped to the outside by the vacuum pump. The presence of test gas within the chamber results in an increase of the electric current drawn by the vacuum pump if compared to vacuum conditions. The increase of the electric current is signalled by a detector informing of the presence of the test gas and, consequently, of a probable leak in the volume to be tested.

To achieve a good sensitivity, the membrane must be very thin, since gas permeability is inversely proportional to the membrane thickness. Moreover, the membrane must resist to high temperatures, since gas permeability is proportional to the membrane temperature.

The membranes presently used generally consist of a capillary tube and the electrical resistor for heating the membrane is helically wound around the capillary tube. A leak detector having a capillary tube membrane is disclosed for instance in patent application No. EP 0352371 "Helium leak detector with silica glass probe".

Capillary tube membranes however are fragile, and securing the capillary tube to the vacuum line is difficult. Moreover, the capillary tube shape is not satisfactory in terms of sensitivity, since it is impossible to heat the capillary tube surface wholly and uniformly to the ideal temperature for a good permeability to the test gas. This is due in part to the limitations in possibility of increasing the resistor's temperature, and the capillary tube is glued to the vacuum line.

Moreover, the capillary tube shape increases the chamber volume and, consequently, both the response inertia of the detector in the presence of the test gas, and the time necessary to have the detector again operating after a leak has been detected.

Planar membranes have been developed in the past to obviate these drawbacks.

These membranes have a composite structure in which a conventional metallic support layer, providing the structural strength, is associated with a thin layer of a material selectively permeable to the test gas. The support layer, which is of a gas impermeable material, has openings or windows through which the permeable layer is exposed at both faces. An example of such a membrane is disclosed in US patent No. 3,505,180, in which a hydrogen-permeable layer of palladium is superimposed to a metal support layer provided with openings.

Yet, also that solution is not wholly satisfactory because of the different physical properties of the materials forming the membrane. For instance, the different thermal expansion coefficients may compromise the membrane life. Moreover, separation phenomena of the different layers forming the composite structure may occur. The latter drawback is very penalising in terms of permeability to the test gas, since it limits the temperature to which the membrane can be heated.

It is a main object of the present invention to provide a selective membrane for leak detectors, allowing overcoming the above drawbacks, as well as a method for producing such a membrane.

It is another object of the present invention to provide a selective membrane for gas detection, having high sensitivity and reliability.

The above and other objects are achieved by a membrane for gas detection according to the invention, as claimed in the appended claims.

SUMMARY OF THE INVENTION

The membrane of the present invention can be kept at high temperature, without risks of loss of integrity, and hence it provides an extremely sensitive and reliable means for leak detection.

A gas-selective membrane made of a body comprising a material that is permeable to at least one selected test gas and substantially impermeable to at least another gas. At least one reduced thickness area that is highly permeable to the selected test gas is formed on the body by removing the material from the body according to the detailed description of the method of manufacturing of the membrane. This reduced thickness area is surrounded by thicker area at least partly for structural strength of the membrane. The at least one reduced thickness area is heated by electrical resistor that partly covering the reduced thickness area.

An apparatus for gas leak detection having a vacuum-tight chamber with a vacuum pump connected thereto incorporates this gas-selective permeable membrane separating at least a portion of the vacuum chamber from the outside environment.

A non- limiting exemplary embodiment of the membrane according to the invention and of the method of manufacturing thereof is disclosed in the detailed description of the invention with reference to the accompanying drawings.

BRIEF DESCRIPTION OF THE DRAWINGS

Fig. 1 is a plan view of the membrane for gas detection according to the invention;

Fig. 2 is a schematic cross sectional view of the membrane according to the invention;

Fig. 3 is a perspective view of a detail of the membrane;

Fig. 4 is a schematic view of a leak detector equipped with a membrane according to the invention; and

Figs. 5a, 5b, 5c to 5d show the main steps of the method according to the invention for producing the membrane.

DETAILED DESCRIPTION OF THE PREFERRED EMBODIMENT

Referring to Figs. 1 and 2, there is shown a membrane 1 according to the invention, comprising a body 11 in which dead cavities 13 are formed, which define an equal number of reduced thickness areas 15 on membrane 1.

Body 11 of membrane 1 preferably consists of a sheet-like disc and it is made of a material selectively permeable to gases.

For example, quartz, glass with high silica content and palladium are materials selectively permeable to gases.

If membrane 1 is used for detecting helium, the material used for producing the membrane will preferably be quartz or glass with high silica content. In such case, the thickness of membrane 1 will preferably be in the range 800 to 900 μm, and reduced thickness areas 15 will be about 10 μm thick.

Cavities 13 are preferably circular and have in axial direction an outward-flaring conical cross section. Moreover, said cavities 13 will preferably be formed on a same face 11a of membrane 1.

Heating means 17 are provided on the opposite face 11b of membrane 1. An electric resistor adhering to the face 11b of membrane 1 and extending through all reduced thickness areas 15 forms heating means 17.

Advantageously, in order to uniformly heat reduced thickness areas 15, resistor 17 extends along at least a portion of the perimeter of areas 15, preferably according to a circular path that is located substantially at an intermediate distance between the centre of each area 15 and the outer edge thereof.

Thus, areas 15 can be uniformly heated and the temperature required to make the material gas permeable is uniformly obtained over the whole corresponding area 15. Moreover, resistor 17 is equipped with a pair of terminals 19 for connecting resistor 17 to an electric current source (not shown).

Advantageously, according to the invention, both areas 15 and resistor 17 heating them is located within a perimeter defined by an annulus 11c having sufficient width to ensure the effective bonding of membrane 1, for instance by gluing, to the walls of the vacuum-tight chamber of the leak detector. Advantageously, said annulus 11c will be substantially "cold" with respect to areas 15, since it is not run through by resistor 17. Thus, the adhesion of membrane 1 to the chamber walls will not be harmed.

As better shown in Fig. 3, resistor 17 comprises a film 17a of a conductive material, preferably chromium or in the alternative copper or aluminium, and is bonded to membrane 1 through a layer of adhesive material 17b, for example of titanium. Conductive layer 17a is moreover coated with a protecting layer 17c, for instance of gold.

Referring to Fig. 4, there is schematically shown a leak detector, generally denoted 31. Detector 31 comprises a vacuum-tight chamber 33 obtained by means of a hollow cylindrical body 39, one end of which is connected to the suction port of a vacuum pump 37, for example, an ionic pump. The other end of chamber 33 is separated from the outside environment by a gas-selective permeable membrane 1, of the kind described with reference to the previous Figures.

Advantageously, said membrane 1 is bonded to cylindrical body 39 defining chamber 33 along circular rim 41 of said cylindrical body 39. Membrane 1 is preferably bonded to said rim

41 by gluing peripheral annulus 11c of membrane 1.

In the alternative, membrane 1 may be glued to a metal ring, subsequently brazed to rim 41 of chamber 33.

Membrane 1 is preferably mounted so that electric resistor 17 faces the outside of chamber 33.

Moreover, reduced thickness areas 15 are so distributed that annulus 11c of the membrane, attached by the gluing to rim 41, is kept at a sufficiently low temperature in order not to harm the holding of the gluing.

The apparatus thus obtained is placed in the environment to be tested, into which a certain amount of test gas might have been previously introduced. An electronic supply unit 19 connected to pump 17 is arranged to detect the presence of test gas, if any, inside chamber 33 thanks to the variation in the current drawn by the pump.

Referring to Figs. 5a to 5d, the major steps of the method of manufacturing a gas-selective permeable membrane are shown.

First, as shown in Fig. 5a, a sheet 51 of a material selectively permeable to the test gas, for instance amorphous quartz, is coated with a uniform layer of amorphous silicon 53. A thin uniform layer 55 of a photosensitive material, (for instance the commercially available material Photoresist HPR504 ARCH Positive) is applied onto layer 53. Subsequently layer 55 is covered with a lithographic mask 57 having openings 59 in correspondence with the areas of sheet 51 where a reduced thickness is to be obtained. Said mask 57 may be formed by instance by using chromium deposited on optical quartz, or a polyester film commercially available under the name "Mylar®".

The above assembly is exposed to ultra-violet radiation UV perpendicular to sheet 51, on the side where lithographic mask 57 is provided.

The effect of radiation is to remove material from photosensitive layer 55 in the exposed areas, i.e. in the areas corresponding to openings 59 in mask 57. Thus the pattern of openings 59 in mask 57 is reproduced on photosensitive layer 55.

At the end of the irradiation step, lithographic mask 57 is removed and sheet 51 is submitted to dry etching by means of a plasma, preferably of CF_4, as shown in Fig. 5b. Plasma etching only affects amorphous silicon layer 53 in the exposed areas corresponding to openings 61 in photosensitive layer 55, so that the pattern of the openings in photosensitive layer 55 is reproduced on amorphous silicon layer 53.

Photosensitive layer 55 is then removed and sheet 51 is submitted to drilling by a ultrasonic drill 63, as shown in Fig. 5c. Ultrasonic drilling only provided within the areas in sheet 51 that are left uncovered by amorphous silicon layer 53, in correspondence with openings

65, and creates a plurality of cavities 13 in sheet 51: thus, an equal number of reduced thickness areas 15, highly permeable to the test gas, will be defined.

A further step of the method according to the invention, shown in Fig. 5d, is a wet etching treatment. Sheet 51, still partly coated with amorphous silicon layer 53, is placed into a suitable cell 71, suspended by means of a frame 73 on which sheet 51 is placed while being supported by ring seals 37. Sheet 51 is immersed into a bath 75 of HF and water, by the action of which cavities 13 are finished by wet etching.

Once the processing of the membrane is complete, amorphous silicon layer 53 is removed and, if necessary, the heating resistor is applied.

According to another embodiment, the method of manufacturing the membrane is achieved by directly treating a sheet of a material selectively permeable to the test gas, for instance amorphous quartz, by ultrasounds in order to obtain a plurality of reduced thickness areas. According to this method, ultrasonic drills of extremely high precision should be utilised.